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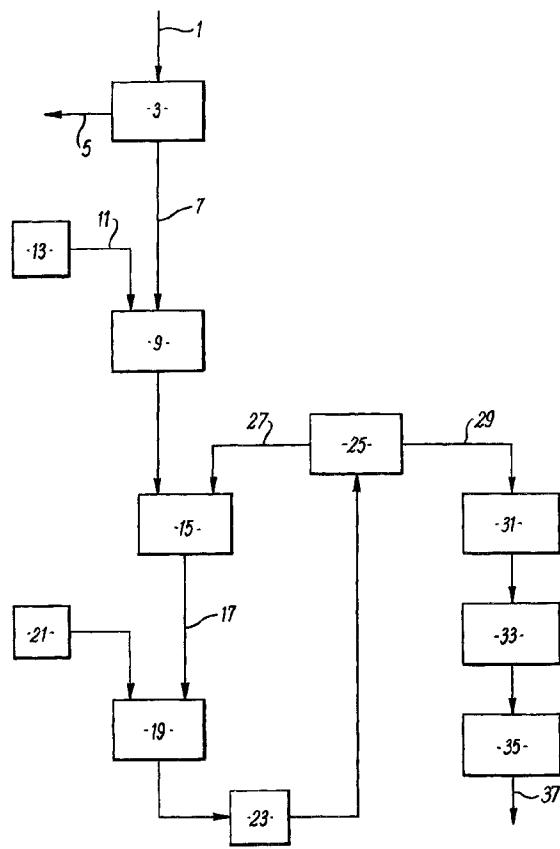
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[Continued on next page]

(54) Title: KAOLIN PRODUCTS AND THEIR PRODUCTION



**WO 02/16511 A1**



(57) Abstract: A method of producing a kaolin particulate material suitable for use as improved SC filler includes the steps of: (a) preparing an aqueous feed suspension of kaolin; (b) treating the kaolin of the feed suspension by attrition grinding in a grinding vessel using grinding media granules; (c) delivering an aqueous suspension of the kaolin ground in step (b) to a particle size classifier and separating the kaolin of the suspension into a coarse fraction and a fine fraction; (d) returning an aqueous suspension of the coarse fraction produced in step (c) to the grinding vessel to be further attrition ground; and (e) extracting an aqueous suspension of the fine fraction produced in step (c) as a product suspension; wherein the kaolin of the feed suspension in step (a) is such that not more than 30 % by weight has a particle size less than 2  $\mu\text{m}$  and not more than 30 % by weight has a particle size greater than 10  $\mu\text{m}$  and wherein the kaolin of the product suspension produced in step (e) is such that not more than 10 % by weight has a particle size greater than 10  $\mu\text{m}$  and not more than 10 % by weight has a particle size less than 0.25  $\mu\text{m}$ .



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#### KAOLIN PRODUCTS AND THEIR PRODUCTION

The present invention relates to kaolin products and their production. In particular, it relates to 5 kaolin fillers for use in making super-calendered (SC) papers and their production.

Mineral fillers are used in the production of most papers for printing or writing applications. The incorporation of such fillers together with 10 cellulose fibres in a paper making composition reduces the overall cost of the raw materials and can improve optical and printing properties of the paper. However, adding fillers causes reduction in the strength of the paper, so there is a practical limit 15 to the amount of fillers normally used in the paper making composition.

Highly filled uncoated papers can compete with some coated paper grades. Suitable smoothness, gloss, density per unit area and printing ink porosity can 20 be achieved by multiple calendering of the filled paper sheets. The well known process of calendering involves compressing the sheets between rollers in one or more passes (each pass being known in the art as a 'nip').

Highly filled uncoated, calendered papers having 25 properties approaching those of coated papers are generally known as super-calendered (SC) papers. Such papers can be used for various applications, particularly involving printing upon the paper by 30 rotogravure or offset processes.

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Kaolin materials have been widely used in the prior art as fillers for making SC papers. One purpose of the present invention is to provide hydrous kaolin materials suitable for use as improved 5 fillers in the production of SC papers. Another purpose is to provide an improved method of making such materials.

According to the present invention in a first aspect there is provided a method of producing a 10 kaolin particulate material suitable for use as improved SC filler includes the steps of:

- (a) preparing an aqueous feed suspension of kaolin;
- (b) treating the kaolin of the feed suspension by attrition grinding in a grinding vessel using 15 grinding media granules;
- (c) delivering an aqueous suspension of the kaolin ground in step (b) to a particle size classifier and separating the kaolin of the suspension into a coarse fraction and a fine fraction;
- (d) returning an aqueous suspension of the coarse fraction produced in step (c) to the grinding vessel 20 to be further attrition ground; and
- (e) extracting an aqueous suspension of the fine fraction produced in step (c) as a product 25 suspension;

wherein the kaolin of the feed suspension in step (a) is such that not more than 30% by weight has a particle size less than 2 $\mu$ m and not more than 30% by weight has a particle size greater than 10 $\mu$ m and 30 wherein the kaolin of the product suspension produced in step (e) is such that not more than 10% by weight

has a particle size greater than 10 $\mu\text{m}$  and not more than 10% by weight has a particle size less than 0.25 $\mu\text{m}$ .

The required particle size distribution of the  
5 kaolin feed suspension may be obtained by treating a  
pre-feed aqueous suspension of kaolin by  
classification using a suitable particle size  
classifier and selecting the coarse fraction produced  
by the classifier as the feed suspension in step (a).  
10 The classifier may comprise a centrifuge, e.g. a  
decanter centrifuge.

Preferably, in step (a) not more than 20% by  
weight of the particles have a particular size of  
less than 2 $\mu\text{m}$ . Preferably, in step (a) not more than  
15 25%, desirably less than 20%, by weight of the  
particles have a size greater than 10 $\mu\text{m}$ .

In the grinding step (b) in the method according  
to the first aspect of the invention, the grinding  
may be carried out in a vertical stirred grinding  
20 vessel of the kind described in GB1469028. The work  
input applied in the grinding vessel in step (b) may  
for example be in the range of from 10kw.h.t<sup>-1</sup> to 300  
kw.h.t<sup>-1</sup> particularly from 25kw.h.t<sup>-1</sup> to 100kw.h.t<sup>-1</sup>  
based on the dry weight of kaolin processed.

25 The solids content of the suspension of kaolin  
during grinding may be selected from a wide range of  
solids contents to give dilute, medium solids or  
concentrated suspensions. We prefer to use a solids  
content of from 15% to 35% by weight during grinding.  
30 A dispersant, e.g. one of the kaolin dispersants well  
known in the art, such as an organic agent, e.g.

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sodium polyacrylate, or an inorganic agent, e.g. sodium hexametaphosphate, may be present during grinding.

Preferably, during grinding, the pH of the 5 suspension is in the range 4 to 7, especially from 5 to 7.

Preferably, the granules of the grinding media are such that at least 90% by weight of the granules have sizes in the range 1mm to 2mm.

10 The particle size classifier used in step (c) may comprise one or more hydrocyclones or one or more decanter centrifuges or a combination of one or more hydrocyclones and/or more decanter centrifuges. For example, where multiple devices are used, these may 15 be arranged in parallel, a portion of the suspension to be treated being fed through each device.

20 The solids content of the suspension delivered to the particle size classifier in step (c) may be reduced after the suspension leaves the grinding vessel in which the suspension is ground prior to delivery to the particle size classifier. Water may be added to the suspension which leaves the grinding vessel to dilute the suspension delivered to the particle size classifier. For example, the solids 25 content may be reduced by a differential percentage by weight of between 5% and 20% by weight, e.g. by between 5% and 10% by weight. The solids content of the coarse fraction returned to the grinding vessel in step (d) may be greater than that of the 30 suspension in the grinding vessel in step (b). For example, the solids content of the returned coarse

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fraction may be in the range 25% to 40% by weight. The solids content of the returned coarse fraction may be increased by a differential percentage by weight of at least 2%, e.g. by between 5% and 20% by 5 weight compared with that in the grinding vessel in step (b).

We have found that the method according to the first aspect of the invention unexpectedly and beneficially allows kaolin particulate materials to 10 be made which show improved properties when used as fillers to be produced in an economical manner. Preferably, the method according to the first aspect of the invention includes no stages involving separation of impurities by chemical treatment, e.g. 15 froth flotation. Avoiding such stages enables an economic production process to be achieved.

Kaolin of the product suspension extracted in the step (e) may be subjected to one or more further known treatment steps, e.g. screening, pH adjustment, 20 ageing to thicken and/or condition and bleaching. The suspension is desirably dewatered, e.g. by one or more techniques such as filtering or pressing and/or thermally dried to a powder.

The kaolin of the product suspension extracted 25 in step (e), herein the 'product kaolin', may have the following properties. The cumulative percentages P by weight of particles of the product kaolin having a size greater or less than X for different values of X are preferably as follows.

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X (μm)	P (%)
>10	Not more than 10
<2	Not less than 30
<1	Not less than 15
<0.25	Not more than 10

Preferably, the cumulative percentages P of particles of the product kaolin having a size greater or less than X for different values of X are as follows:

X (μm)	P (%)
>10	Not more than 7
<2	Not less than 33
<1	Not less than 20
<0.25	Less than 10

In some cases, the cumulative percentages P of particles of the product kaolin having a size greater or less than X for different values of X are as follows:

X (μm)	P (%)
>10	Not more than 7
<2	At least 60
<1	At least 40
<0.5	At least 15
<0.25	Less than 10

In this specification, all particle size properties including particle size distributions or

plots of P versus X as described above and also mean particle size are as determined for a fully dispersed dilute aqueous suspension of the particulate material in question by sedimentation using a SEDIGRAPH™ 5100 machine, (supplied by the Micromeritics Corporation) in a well-known manner.

10 The mean particle size of the kaolin product (value of X defined above when P is 50%) may be from 0.5 $\mu$ m to 2.0 $\mu$ m, e.g. from 0.9 $\mu$ m to 1.6 $\mu$ m.

15 The Einlehener abrasion of the kaolin product may be less than 90g.m<sup>-2</sup>, preferably not greater than than 80g.m<sup>-2</sup>

20 The steepness factor of the kaolin product may be at least 32, in some cases at least 35 or preferably 40 or higher. The 'steepness factor', (sometimes referred to as 'narrowness') refers to the steepness of the particle size distribution curve as measured by the SEDIGRAPH 5100 machine in the manner described earlier and is given by the expression 100x(d<sub>30</sub>÷d<sub>70</sub>), where d<sub>30</sub> is the value of the particle size less than which there are 30% by weight of the particles and d<sub>70</sub> is the value of the particle size less than which there are 70% by weight of the particles.

25 The brightness of the kaolin product may be at least 80 ISO units, preferably at least 82 ISO units measured in a well-known manner according industry (TAPPI) standard procedures.

30 The shape factor of the particles of the kaolin product may be at least 30, preferably at least 40, in some cases at least 50. The expression 'shape

factor' as used herein means the average value (on a weight average basis) of the ratio of mean particle diameter to particle thickness for a population of particles ie the kaolin product of varying size and 5 shape as measured using the electrical conductivity method and apparatus described in GB-A-2240398, US-A-5128606 and EP-A-528078 and using the equations derived in these patent specifications. 'Mean particle diameter' is here defined as the diameter of 10 a circle, which has the same area as the largest face of the particle. In the electrical conductivity measurement method described in the said specifications, the electrical conductivity of a fully dispersed aqueous suspension of the particles 15 under test is caused to flow through an elongated tube. Measurements of the electrical conductivity are taken between (a) a pair of electrodes separated longitudinally along the axis of the tube and (b) a pair of electrodes separated transversely across the 20 tube. The shape factor of the particles under test calculated from the two conductivity measurements.

Kaolin products having the properties described earlier, obtained by use of the method according to the first aspect of the invention in their 25 production, show improved performance as SC fillers. In particular, the combination of the defined values of steepness factor and shape factor give, in an SC paper made from the kaolin product, a beneficially enhanced combination of high brightness and high 30 porosity and thereby enhanced printability,

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especially for printing by the rotogravure or offset process.

According to the present invention in a second aspect there is provided a composition for use in 5 making paper to be treated by a super-calendering process includes an aqueous suspension of cellulose fibres and filler particles, wherein the filler particles comprise the kaolin product defined earlier.

10 The filler particles may constitute at least 10%, preferably up to 40%, of the solids content of the composition according to the second aspect.

The kaolin product may form substantially all of the said filler particles or may be blended in a 15 known manner with other filler particles in the said composition. For example, calcium carbonate, which may be particulate material either obtained from natural sources and processed for example by grinding or which has been chemically synthesised 20 (precipitated) may be included in such a blend on account of their brightness contribution. For example, the filler particles may comprise from 60% to 100% by weight of the kaolin product and from 0% to 40% by weight calcium carbonate. Other filler 25 materials, which may optionally be included in a blend of materials together with the product kaolin include other hydrous kaolin, calcined kaolin,  $TiO_2$ , calcium sulphate or other fillers well known in the paper making art.

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Embodiments of the present invention will now be described by way of example with reference to the accompanying drawing, in which:

Figure 1 is a diagram in schematic flowsheet 5 form of an arrangement of apparatus for treating kaolin in accordance with a method embodying the invention.

As shown in Figure 1, a pre-feed dispersed kaolin suspension 1 is fed to a decanter centrifuge 3 10 in which it is separated into a fine fraction 5 and a coarse fraction 7. The fine fraction 5 may be further processed and used as a product in a known manner. The pH of the coarse fraction is adjusted to about pH5 in a vessel 9 by addition of alkali 11 from a 15 source 13. The kaolin of the resulting suspension has a particle size distribution such that not greater than 20% by weight of the kaolin particles have a particle size of greater than 10 $\mu\text{m}$ , not greater than 20% by weight have a particle size of less than 2 $\mu\text{m}$  20 and less than 5% by weight have a particle size of less than 0.25 $\mu\text{m}$ . This kaolin is delivered as a feed suspension to a media attrition grinder 15 of the kind described earlier. The suspension is ground in the grinder 15.

25 A suspension 17 of ground kaolin from the grinder 15 is fed to a mixing vessel 19 where it is diluted by addition of water from a source 21. The diluted suspension is pumped by a pump 23 to a bank 25 of hydrocyclones through which portions of the 30 suspension are fed and treated. The hydrocyclones separate the kaolin into a coarse fraction 27 and a

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fine fraction 29. The course fraction 27, which has a solids content greater than 30%, is returned to the grinder 15. The fine fraction 29 is further treated in a known manner by processing 31, e.g. screening to 5 remove large particles, pH adjustment to pH 4, and bleaching using a dithionite reductive bleaching agent. Finally, the fine fraction 29 is dewatered by a tube press filter 33 to a moisture content of about 18% by weight and, if desired in dry form, thermally 10 dried in a drier 35 to produce a final kaolin product 37.

Examples of kaolin products produced in the manner described above with reference to Figure 1 are as follows.

15

Example 1

A first example of a kaolin product embodying the invention showed the following properties (where X 20 and P are as defined earlier):

X (μm)	P (%)
>10	Not more than 6
<2	38
<1	24
<0.5	13
<0.25	Not more than 7

The kaolin product also had the following properties:

Powder brightness (ISO units): 80

25 Yellowness (ISO units): 4.0

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Abrasion: 85mg.m<sup>-2</sup>

Shape factor: 30

Example 2

5

A second example of a kaolin product embodying the invention showed the following properties (where X and P are as defined earlier):

X (μm)	P (%)
>5	15
<2	50
<1	30
<0.25	Not more than 8

10

The kaolin product also had the following properties:

Powder brightness (ISO units): 82.5

Yellowness (ISO units): 5.0

Abrasion 75 g.m<sup>-2</sup>

15

Shape factor: 45

## CLAIMS

1. A method of producing a kaolin particulate material suitable for use as improved SC filler including the steps of:
  - 5 (a) preparing an aqueous feed suspension of kaolin ;
  - (b) treating the kaolin of the feed suspension by attrition grinding in a grinding vessel using grinding media granules;
  - 10 (c) delivering an aqueous suspension of the kaolin ground in step (b) to a particle size classifier and separating the kaolin of the suspension into a coarse fraction and a fine fraction;
  - (d) returning an aqueous suspension of the coarse
  - 15 fraction produced in step (c) to the grinding vessel to be further attrition ground; and
  - (e) extracting an aqueous suspension of the fine fraction produced in step (c) as a product suspension;
- 20 wherein the kaolin of the feed suspension in step (a) is such that not more than 30% by weight has a particle size less than 2 $\mu$ m and not more than 30% by weight has a particle size greater than 10 $\mu$ m and wherein the kaolin of the product suspension produced
- 25 in step (e) is such that not more than 10% by weight has a particle size greater than 10 $\mu$ m and not more than 10% by weight has a particle size less than 0.25 $\mu$ m.
2. A method according to claim 1 and wherein the
- 30 kaolin feed suspension employed in step (a) is a

coarse fraction obtained from a particle size classifier.

3. A method according to claim 1 or 2 and wherein not more than 20 per cent by weight of the particles 5 of the kaolin in step (a) have a size less than 2 $\mu$ m.
4. A method according to claim 1, 2 or 3 and wherein not more than 25 per cent by weight of the kaolin particles in step (a) have a size greater than 10 $\mu$ m.
5. A method according to any one of the preceding 10 claims and wherein in step (c) the particle size classifier comprises one or more hydrocyclones.
6. A method according to claim 5 and wherein the particle size classifier comprises a plurality of hydrocyclones arranged in parallel.
- 15 7. A method according to any one of the preceding claims and wherein water is added to the suspension which leaves the grinding vessel prior to delivery to the particle size classifier employed in step (c).
8. A method according to claim 7 and wherein the 20 solids content of the suspension is reduced by a differential percentage amount of from 5% to 20% by weight by the water addition.
9. A method according to any one of the preceding claims and wherein the solids content of the coarse 25 fraction returned to the grinding vessel in step (e) is a differential percentage of at least 2% by weight greater than the solids content of the suspension in the grinding vessel in step (b).
10. A product kaolin for use as a filler for super- 30 calendered paper which comprises kaolin produced from the fine fraction suspension extracted in step (e) of

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the method according to any one of the preceding claims.

11. A product kaolin according to claim 10 and wherein the product kaolin has a particle size  
 5 distribution such that the cumulative percentages P by weight of particles of the product kaolin having a size greater or less than X for different values of X are as in the following table:

X ( $\mu\text{m}$ )	P (%)
>10	Not more than 10
<2	Not less than 30
<1	Not less than 15
<0.25	Not more than 10

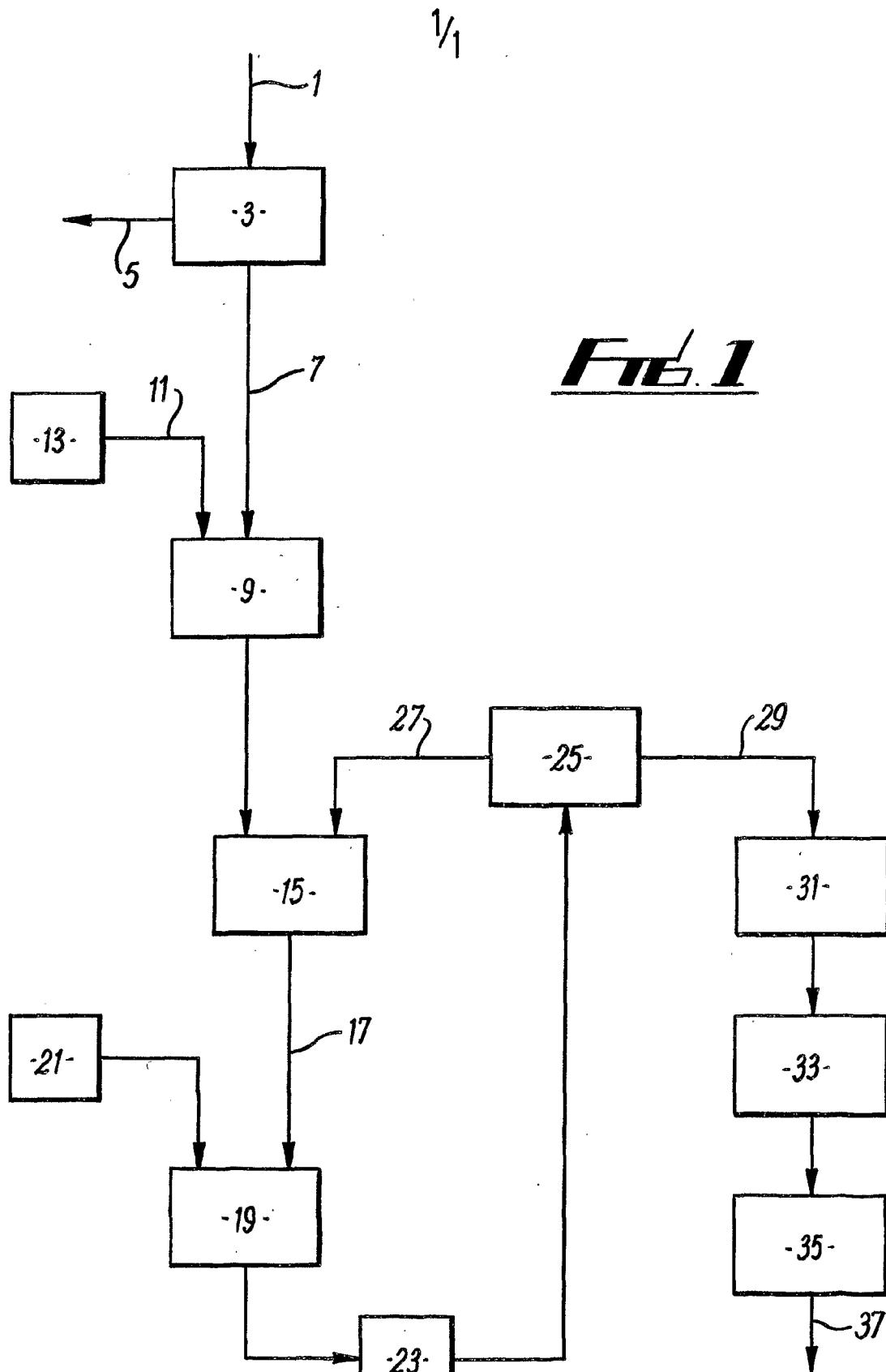
10 12. A product kaolin according to claim 10 and wherein the product kaolin is such that the cumulative percentages P of particles of the product kaolin having a size greater or less than X for  
 15 different values of X are as follows:

X ( $\mu\text{m}$ )	P (%)
>10	Not more than 7
<2	Not less than 33
<1	Not less than 20
<0.25	Less than 10

13. A product kaolin according to claim 11 and wherein the product kaolin is such that the cumulative percentages P of particles of the product  
 20 kaolin having a size greater or less than X for different values of X are as in the following table:

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X (μm)	P (%)
>10	Not more than 7
<2	At least 60
<1	At least 40
<0.5	At least 15
<0.25	Less than 10



# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/GB 01/03717

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7 C09C1/42

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
IPC 7 C09C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, CHEM ABS Data, WPI Data, PAJ

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 99 51815 A (HUSBAND JOHN CLAUDE ; ECC INT LTD (GB); PAYTON DESMOND CHARLES (GB)) 14 October 1999 (1999-10-14) page 4, line 22 -page 5, line 3 page 9, line 7 -page 10, line 22 page 11, line 18 -page 14, line 12 example 1 ---	1-5,9-13
A	US 4 948 664 A (BROCINER RONALD E) 14 August 1990 (1990-08-14) column 5, line 3 -column 7, line 65 ---	10-15
A	WO 98 58613 A (PERSSON CHARLOTTE ; MOELENLYCKE AB (SE); SAMUELSSON ANN (SE)) 30 December 1998 (1998-12-30) page 2, line 27 -page 4, line 26 page 13, line 8 -page 14, line 24; claim 1; examples 1,2 ---	1-15
	-/-	

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

° Special categories of cited documents :

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**INTERNATIONAL SEARCH REPORT**

International Application No  
PCT/GB 01/03717

**C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT**

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A,P	WO 00 59840 A (HUSBAND JOHN CLAUDE ;IMERYS PIGMENTS INC (US); BROWN HARRY VINCENT) 12 October 2000 (2000-10-12) page 7, line 15 -page 11, line 26; claims 11,12; example 1 -----	1-15

**INTERNATIONAL SEARCH REPORT**

Information on patent family members

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